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## 4-Methoxy-3-(4-nitrobenzyloxy)-benzaldehyde

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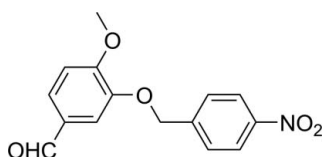
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Key indicators: single-crystal X-ray study;  $T = 294$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å;  $R$  factor = 0.057;  $wR$  factor = 0.133; data-to-parameter ratio = 16.5.

In the title compound,  $\text{C}_{15}\text{H}_{13}\text{NO}_5$ , the two benzene rings make a dihedral angle of  $3.98(7)^\circ$ . The crystal packing is stabilized by weak non-classical intermolecular  $\text{C}-\text{H}\cdots\text{O}$  interactions that link molecules into centrosymmetric tetramers.

## Related literature

For general background to the use of Schiff base derivatives in the development protein and enzyme mimics, see: Santos *et al.* (2001). For a closely related crystal structure, see: Li & Chen (2008). For reference bond-length data, see: Allen *et al.* (1987).



## Experimental

## Crystal data

$\text{C}_{15}\text{H}_{13}\text{NO}_5$   
 $M_r = 287.26$   
 Monoclinic,  $P2_1/c$   
 $a = 6.853(1)$  Å  
 $b = 11.994(2)$  Å

$c = 16.405(3)$  Å  
 $\beta = 98.28(3)^\circ$   
 $V = 1334.4(4)$  Å<sup>3</sup>  
 $Z = 4$   
 Mo  $K\alpha$  radiation

$\mu = 0.11$  mm<sup>-1</sup>  
 $T = 294$  K

0.22 × 0.16 × 0.11 mm

## Data collection

Bruker SMART APEX CCD area-detector diffractometer  
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)  
 $T_{\min} = 0.932$ ,  $T_{\max} = 0.988$

10078 measured reflections  
 3161 independent reflections  
 2441 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.047$

## Refinement

$R[F^2 > 2\sigma(F^2)] = 0.057$   
 $wR(F^2) = 0.133$   
 $S = 1.12$   
 3161 reflections

191 parameters  
 H-atom parameters constrained  
 $\Delta\rho_{\text{max}} = 0.26$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.21$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

| $D-\text{H}\cdots A$                               | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|--|--------------|--------------------|-------------|----------------------|
| $\text{C12}-\text{H12}\cdots\text{O3}^{\text{i}}$  | 0.93         | 2.42               | 3.280 (2)   | 154                  |
| $\text{C9}-\text{H9A}\cdots\text{O5}^{\text{ii}}$  | 0.97         | 2.53               | 3.383 (2)   | 147                  |
| $\text{C8}-\text{H8B}\cdots\text{O4}^{\text{iii}}$ | 0.96         | 2.55               | 3.410 (3)   | 150                  |

Symmetry codes: (i)  $x, -y - \frac{1}{2}, z + \frac{1}{2}$ ; (ii)  $x, -y - \frac{1}{2}, z - \frac{1}{2}$ ; (iii)  $-x + 1, y + \frac{1}{2}, -z + \frac{3}{2}$ .

Data collection: SMART (Bruker, 1999); cell refinement: SAINT (Bruker, 1999); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); software used to prepare material for publication: SHELXTL.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WN2430).

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**supplementary materials**

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## 4-Methoxy-3-(4-nitrobenzyloxy)benzaldehyde

Z.-Y. Duan, G.-L. Ma and L.-P. Yang

### Comment

Many Schiff base derivatives have been synthesized and employed to develop protein and enzyme mimics (Santos *et al.*, 2001). The synthesis and crystal structures of numerous derivatives have been published. In particular, the isomeric 3-methoxy-4-(4-nitrobenzyloxy)benzaldehyde crystal structure has been reported (Li & Chen, 2008). As a part of our interest in the coordination properties of Schiff bases functioning as ligands, we have investigated the title compound, which has been used as a precursor in the preparation of Schiff bases.

In the title molecule (Fig. 1), bond lengths (Allen *et al.*, 1987) and angles are within normal ranges. The two benzene rings make a dihedral angle of 3.98 (7)° with each other. A similar value of 4.99 (6)° is observed in 3-methoxy-4-(4-nitrobenzyloxy)benzaldehyde (Li & Chen, 2008).

The crystal packing is stabilised by weak, non-classical intermolecular C12—H12...O3=C7, C8—H8B...O4 and C9—H9A...O5 interactions that link adjacent molecules into centrosymmetric tetramers (Table 1, Fig. 2).

### Experimental

An anhydrous acetonitrile solution (100 ml) of 3-hydroxy-4-methoxybenzaldehyde (1.52 g, 10 mmol) was added dropwise to a solution (50 ml) of 1-(bromomethyl)-4-nitrobenzene (2.16 g, 10 mmol) and pyridine (0.79 g, 10 mmol) in acetonitrile, over a period of 30 min., and the mixture refluxed for 24 h under a nitrogen atmosphere. The solvent was removed and the resultant mixture poured into ice-water (100 ml). The yellow precipitate was then isolated and recrystallized from acetonitrile. It was then dried in a vacuum to give the pure compound in 78% yield. Pale-yellow single crystals of the title compound suitable for X-ray analysis were obtained by slow evaporation of an acetonitrile solution.

### Refinement

The H atoms were included at calculated positions and refined using a riding model approximation. Constrained C—H bond lengths and isotropic U parameters: 0.93 Å and  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$  for  $C_{\text{sp}^2}$ —H; 0.97 Å and  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$  for methylene C—H; 0.96 Å and  $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$  for methyl C—H.

### Figures

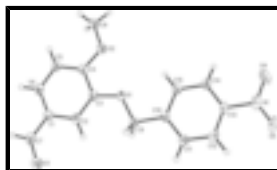


Fig. 1. The molecular structure of the title compound, with displacement ellipsoids for non-H atoms drawn at the 50% probability level. Hydrogen atoms are shown as spheres of arbitrary radius.

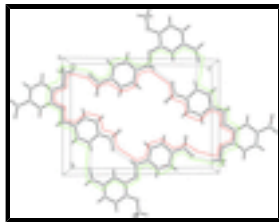


Fig. 2. A packing diagram of the crystal structure, with H bonds drawn as dashed lines. The tetramers are indicated by red and green lines.

## 4-Methoxy-3-(4-nitrobenzyloxy)benzaldehyde

### Crystal data

|                                |   |
|--------------------------------|---|
| $C_{15}H_{13}NO_5$             | $F(000) = 600$  |
| $M_r = 287.26$                 | $D_x = 1.430 \text{ Mg m}^{-3}$                         |
| Monoclinic, $P2_1/c$           | Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$ |
| Hall symbol: -P 2ybc           | Cell parameters from 3519 reflections                   |
| $a = 6.853 (1) \text{ \AA}$    | $\theta = 2.3\text{--}26.2^\circ$                       |
| $b = 11.994 (2) \text{ \AA}$   | $\mu = 0.11 \text{ mm}^{-1}$                            |
| $c = 16.405 (3) \text{ \AA}$   | $T = 294 \text{ K}$                                     |
| $\beta = 98.28 (3)^\circ$      | Block, pale-yellow                                      |
| $V = 1334.4 (4) \text{ \AA}^3$ | $0.22 \times 0.16 \times 0.11 \text{ mm}$               |
| $Z = 4$                        |   |

### Data collection

|   |  |
|---|--|
| Bruker SMART APEX CCD area-detector diffractometer          | 3161 independent reflections   |
| Radiation source: fine-focus sealed tube graphite           | 2441 reflections with $I > 2\sigma(I)$                                 |
| $\varphi$ and $\omega$ scans                                | $R_{\text{int}} = 0.047$   |
| Absorption correction: multi-scan (SADABS; Sheldrick, 1996) | $\theta_{\text{max}} = 27.9^\circ$ , $\theta_{\text{min}} = 2.1^\circ$ |
| $T_{\text{min}} = 0.932$ , $T_{\text{max}} = 0.988$         | $h = -8 \rightarrow 9$   |
| 10078 measured reflections                                  | $k = -14 \rightarrow 15$   |
|   | $l = -19 \rightarrow 21$   |

### Refinement

|                                 |  |
|---------------------------------|--|
| Refinement on $F^2$             | Primary atom site location: structure-invariant direct methods |
| Least-squares matrix: full      | Secondary atom site location: difference Fourier map           |
| $R[F^2 > 2\sigma(F^2)] = 0.057$ | Hydrogen site location: inferred from neighbouring sites       |
| $wR(F^2) = 0.133$               | H-atom parameters constrained                                  |
| $S = 1.12$                      | $w = 1/[\sigma^2(F_o^2) + (0.0561P)^2 + 0.2245P]$              |
| 3161 reflections                | where $P = (F_o^2 + 2F_c^2)/3$                                 |
| 191 parameters                  | $(\Delta/\sigma)_{\text{max}} < 0.001$                         |
| 0 restraints                    | $\Delta\rho_{\text{max}} = 0.26 \text{ e \AA}^{-3}$            |
|                                 | $\Delta\rho_{\text{min}} = -0.21 \text{ e \AA}^{-3}$           |

*Special details*

**Geometry.** All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

**Refinement.** Refinement of  $F^2$  against ALL reflections. The weighted  $R$ -factor  $wR$  and goodness of fit  $S$  are based on  $F^2$ , conventional  $R$ -factors  $R$  are based on  $F$ , with  $F$  set to zero for negative  $F^2$ . The threshold expression of  $F^2 > \sigma(F^2)$  is used only for calculating  $R$ -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement.  $R$ -factors based on  $F^2$  are statistically about twice as large as those based on  $F$ , and  $R$ -factors based on ALL data will be even larger.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

|     | <i>x</i>     | <i>y</i>      | <i>z</i>     | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|---------------|--------------|----------------------------------|
| N1  | 0.3564 (2)   | -0.20291 (14) | 0.78771 (10) | 0.0282 (4)                       |
| O1  | 0.23378 (17) | 0.07084 (9)   | 0.45431 (7)  | 0.0228 (3)                       |
| O2  | 0.27616 (18) | 0.28142 (10)  | 0.46497 (8)  | 0.0261 (3)                       |
| O3  | 0.1082 (2)   | -0.00461 (10) | 0.13474 (9)  | 0.0356 (4)                       |
| O4  | 0.3643 (2)   | -0.13342 (13) | 0.84335 (9)  | 0.0419 (4)                       |
| O5  | 0.3704 (2)   | -0.30380 (11) | 0.79970 (9)  | 0.0387 (4)                       |
| C1  | 0.1427 (2)   | 0.14338 (14)  | 0.23296 (11) | 0.0216 (4)                       |
| C2  | 0.1670 (2)   | 0.07594 (14)  | 0.30415 (11) | 0.0204 (4)                       |
| H2  | 0.1562       | -0.0012       | 0.2995       | 0.024*                           |
| C3  | 0.2070 (2)   | 0.12507 (14)  | 0.38043 (11) | 0.0199 (4)                       |
| C4  | 0.2282 (2)   | 0.24204 (14)  | 0.38720 (11) | 0.0214 (4)                       |
| C5  | 0.2003 (3)   | 0.30806 (14)  | 0.31727 (12) | 0.0241 (4)                       |
| H5A | 0.2104       | 0.3852        | 0.3218       | 0.029*                           |
| C6  | 0.1573 (2)   | 0.25824 (14)  | 0.24028 (11) | 0.0237 (4)                       |
| H6  | 0.1380       | 0.3024        | 0.1932       | 0.028*                           |
| C7  | 0.1090 (3)   | 0.09431 (15)  | 0.15046 (12) | 0.0274 (4)                       |
| H7  | 0.0862       | 0.1432        | 0.1061       | 0.033*                           |
| C8  | 0.3346 (3)   | 0.39608 (15)  | 0.47346 (13) | 0.0326 (5)                       |
| H8A | 0.4333       | 0.4111        | 0.4389       | 0.049*                           |
| H8B | 0.3874       | 0.4110        | 0.5298       | 0.049*                           |
| H8C | 0.2221       | 0.4430        | 0.4573       | 0.049*                           |
| C9  | 0.2195 (2)   | -0.04779 (13) | 0.45450 (11) | 0.0196 (4)                       |
| H9A | 0.3148       | -0.0800       | 0.4229       | 0.024*                           |
| H9B | 0.0886       | -0.0709       | 0.4298       | 0.024*                           |
| C10 | 0.2599 (2)   | -0.08656 (13) | 0.54244 (10) | 0.0174 (3)                       |
| C11 | 0.2435 (2)   | -0.19985 (14) | 0.55914 (11) | 0.0221 (4)                       |
| H11 | 0.2101       | -0.2496       | 0.5159       | 0.027*                           |
| C12 | 0.2765 (2)   | -0.23909 (14) | 0.63929 (12) | 0.0226 (4)                       |
| H12 | 0.2654       | -0.3146       | 0.6505       | 0.027*                           |
| C13 | 0.3264 (2)   | -0.16299 (14) | 0.70222 (11) | 0.0209 (4)                       |
| C14 | 0.3467 (2)   | -0.05042 (14) | 0.68780 (11) | 0.0210 (4)                       |
| H14 | 0.3820       | -0.0011       | 0.7312       | 0.025*                           |
| C15 | 0.3131 (2)   | -0.01262 (14) | 0.60716 (11) | 0.0198 (4)                       |

## supplementary materials

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H15                    0.3263                    0.0629                    0.5962                    0.024\*

### Atomic displacement parameters ( $\text{\AA}^2$ )

|     | $U^{11}$    | $U^{22}$    | $U^{33}$    | $U^{12}$    | $U^{13}$   | $U^{23}$    |
|-----|-------------|-------------|-------------|-------------|------------|-------------|
| N1  | 0.0235 (7)  | 0.0388 (9)  | 0.0219 (9)  | -0.0008 (7) | 0.0020 (6) | 0.0092 (7)  |
| O1  | 0.0325 (7)  | 0.0192 (6)  | 0.0165 (7)  | 0.0005 (5)  | 0.0032 (5) | 0.0041 (5)  |
| O2  | 0.0361 (7)  | 0.0228 (7)  | 0.0194 (7)  | -0.0045 (5) | 0.0035 (5) | -0.0001 (5) |
| O3  | 0.0508 (9)  | 0.0282 (7)  | 0.0270 (8)  | -0.0017 (6) | 0.0027 (7) | -0.0002 (6) |
| O4  | 0.0527 (9)  | 0.0535 (9)  | 0.0186 (8)  | 0.0001 (7)  | 0.0021 (7) | -0.0008 (7) |
| O5  | 0.0439 (8)  | 0.0390 (8)  | 0.0325 (9)  | 0.0001 (6)  | 0.0031 (6) | 0.0191 (7)  |
| C1  | 0.0198 (8)  | 0.0251 (9)  | 0.0200 (9)  | -0.0004 (7) | 0.0036 (7) | 0.0021 (7)  |
| C2  | 0.0184 (8)  | 0.0222 (8)  | 0.0205 (9)  | -0.0006 (6) | 0.0027 (7) | 0.0027 (7)  |
| C3  | 0.0184 (8)  | 0.0226 (9)  | 0.0194 (9)  | 0.0013 (6)  | 0.0048 (7) | 0.0059 (7)  |
| C4  | 0.0190 (8)  | 0.0246 (9)  | 0.0213 (9)  | -0.0002 (6) | 0.0051 (7) | 0.0005 (7)  |
| C5  | 0.0269 (9)  | 0.0210 (8)  | 0.0247 (10) | 0.0006 (7)  | 0.0044 (7) | 0.0041 (7)  |
| C6  | 0.0239 (8)  | 0.0253 (9)  | 0.0221 (10) | 0.0025 (7)  | 0.0045 (7) | 0.0081 (7)  |
| C7  | 0.0303 (10) | 0.0307 (10) | 0.0211 (10) | 0.0016 (8)  | 0.0037 (7) | 0.0060 (8)  |
| C8  | 0.0448 (11) | 0.0232 (9)  | 0.0286 (11) | -0.0065 (8) | 0.0007 (9) | -0.0028 (8) |
| C9  | 0.0209 (8)  | 0.0189 (8)  | 0.0194 (9)  | 0.0004 (6)  | 0.0039 (6) | 0.0016 (7)  |
| C10 | 0.0153 (7)  | 0.0218 (8)  | 0.0159 (9)  | 0.0011 (6)  | 0.0047 (6) | 0.0025 (7)  |
| C11 | 0.0235 (8)  | 0.0218 (8)  | 0.0212 (10) | -0.0009 (7) | 0.0037 (7) | -0.0004 (7) |
| C12 | 0.0222 (8)  | 0.0194 (8)  | 0.0264 (10) | 0.0004 (6)  | 0.0037 (7) | 0.0051 (7)  |
| C13 | 0.0174 (8)  | 0.0281 (9)  | 0.0173 (9)  | 0.0029 (7)  | 0.0031 (6) | 0.0066 (7)  |
| C14 | 0.0194 (8)  | 0.0245 (9)  | 0.0186 (9)  | 0.0017 (7)  | 0.0013 (7) | -0.0013 (7) |
| C15 | 0.0186 (8)  | 0.0193 (8)  | 0.0218 (9)  | 0.0010 (6)  | 0.0041 (7) | 0.0018 (7)  |

### Geometric parameters ( $\text{\AA}$ , $^\circ$ )

|          |             |            |           |
|----------|-------------|------------|-----------|
| N1—O5    | 1.228 (2)   | C7—H7      | 0.9300    |
| N1—O4    | 1.232 (2)   | C8—H8A     | 0.9600    |
| N1—C13   | 1.468 (2)   | C8—H8B     | 0.9600    |
| O1—C3    | 1.365 (2)   | C8—H8C     | 0.9600    |
| O1—C9    | 1.4262 (19) | C9—C10     | 1.503 (2) |
| O2—C4    | 1.356 (2)   | C9—H9A     | 0.9700    |
| O2—C8    | 1.433 (2)   | C9—H9B     | 0.9700    |
| O3—C7    | 1.214 (2)   | C10—C15    | 1.391 (2) |
| C1—C6    | 1.385 (2)   | C10—C11    | 1.394 (2) |
| C1—C2    | 1.411 (2)   | C11—C12    | 1.384 (2) |
| C1—C7    | 1.463 (3)   | C11—H11    | 0.9300    |
| C2—C3    | 1.374 (2)   | C12—C13    | 1.383 (3) |
| C2—H2    | 0.9300      | C12—H12    | 0.9300    |
| C3—C4    | 1.413 (2)   | C13—C14    | 1.381 (2) |
| C4—C5    | 1.384 (2)   | C14—C15    | 1.386 (2) |
| C5—C6    | 1.390 (3)   | C14—H14    | 0.9300    |
| C5—H5A   | 0.9300      | C15—H15    | 0.9300    |
| C6—H6    | 0.9300      |            |           |
| O5—N1—O4 | 123.67 (17) | H8A—C8—H8B | 109.5     |

|           |             |             |             |
|-----------|-------------|-------------|-------------|
| O5—N1—C13 | 118.13 (16) | O2—C8—H8C   | 109.5       |
| O4—N1—C13 | 118.20 (15) | H8A—C8—H8C  | 109.5       |
| C3—O1—C9  | 118.49 (13) | H8B—C8—H8C  | 109.5       |
| C4—O2—C8  | 116.89 (14) | O1—C9—C10   | 107.92 (13) |
| C6—C1—C2  | 120.00 (16) | O1—C9—H9A   | 110.1       |
| C6—C1—C7  | 118.69 (16) | C10—C9—H9A  | 110.1       |
| C2—C1—C7  | 121.27 (16) | O1—C9—H9B   | 110.1       |
| C3—C2—C1  | 119.48 (16) | C10—C9—H9B  | 110.1       |
| C3—C2—H2  | 120.3       | H9A—C9—H9B  | 108.4       |
| C1—C2—H2  | 120.3       | C15—C10—C11 | 119.41 (16) |
| O1—C3—C2  | 126.01 (15) | C15—C10—C9  | 121.81 (15) |
| O1—C3—C4  | 113.84 (15) | C11—C10—C9  | 118.78 (15) |
| C2—C3—C4  | 120.13 (16) | C12—C11—C10 | 120.76 (16) |
| O2—C4—C5  | 124.50 (16) | C12—C11—H11 | 119.6       |
| O2—C4—C3  | 115.35 (15) | C10—C11—H11 | 119.6       |
| C5—C4—C3  | 120.15 (17) | C13—C12—C11 | 118.26 (16) |
| C4—C5—C6  | 119.54 (16) | C13—C12—H12 | 120.9       |
| C4—C5—H5A | 120.2       | C11—C12—H12 | 120.9       |
| C6—C5—H5A | 120.2       | C14—C13—C12 | 122.49 (16) |
| C1—C6—C5  | 120.64 (16) | C14—C13—N1  | 118.61 (16) |
| C1—C6—H6  | 119.7       | C12—C13—N1  | 118.90 (16) |
| C5—C6—H6  | 119.7       | C13—C14—C15 | 118.45 (16) |
| O3—C7—C1  | 125.79 (17) | C13—C14—H14 | 120.8       |
| O3—C7—H7  | 117.1       | C15—C14—H14 | 120.8       |
| C1—C7—H7  | 117.1       | C14—C15—C10 | 120.61 (15) |
| O2—C8—H8A | 109.5       | C14—C15—H15 | 119.7       |
| O2—C8—H8B | 109.5       | C10—C15—H15 | 119.7       |

Hydrogen-bond geometry (Å, °)

| <i>D</i> —H $\cdots$ <i>A</i>     | <i>D</i> —H | H $\cdots$ <i>A</i> | <i>D</i> $\cdots$ <i>A</i> | <i>D</i> —H $\cdots$ <i>A</i> |
|-----------------------------------|-------------|---------------------|----------------------------|-------------------------------|
| C12—H12 $\cdots$ O3 <sup>i</sup>  | 0.93        | 2.42                | 3.280 (2)                  | 154                           |
| C9—H9A $\cdots$ O5 <sup>ii</sup>  | 0.97        | 2.53                | 3.383 (2)                  | 147                           |
| C8—H8B $\cdots$ O4 <sup>iii</sup> | 0.96        | 2.55                | 3.410 (3)                  | 150                           |

Symmetry codes: (i)  $x, -y-1/2, z+1/2$ ; (ii)  $x, -y-1/2, z-1/2$ ; (iii)  $-x+1, y+1/2, -z+3/2$ .

Fig. 1

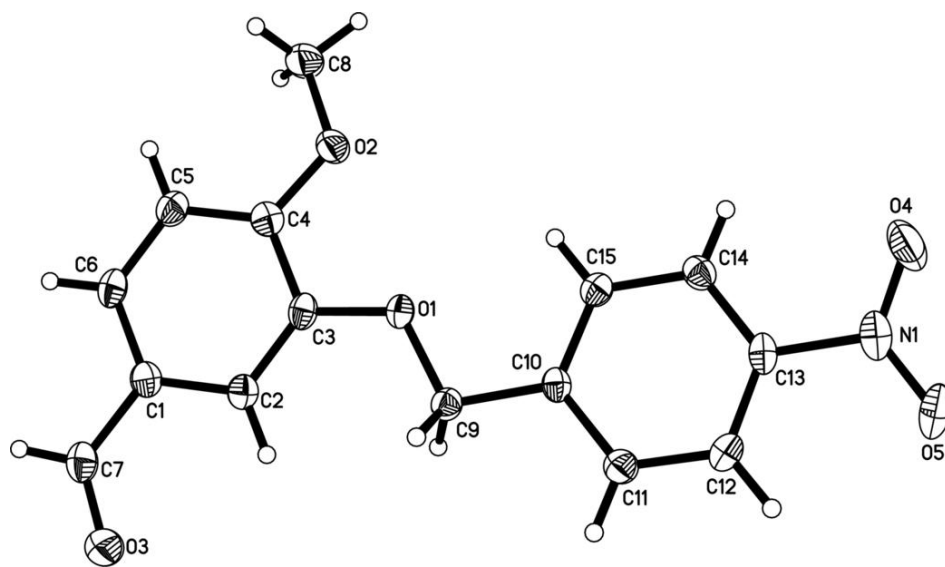




Fig. 2

